



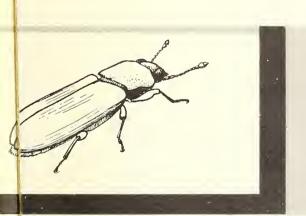
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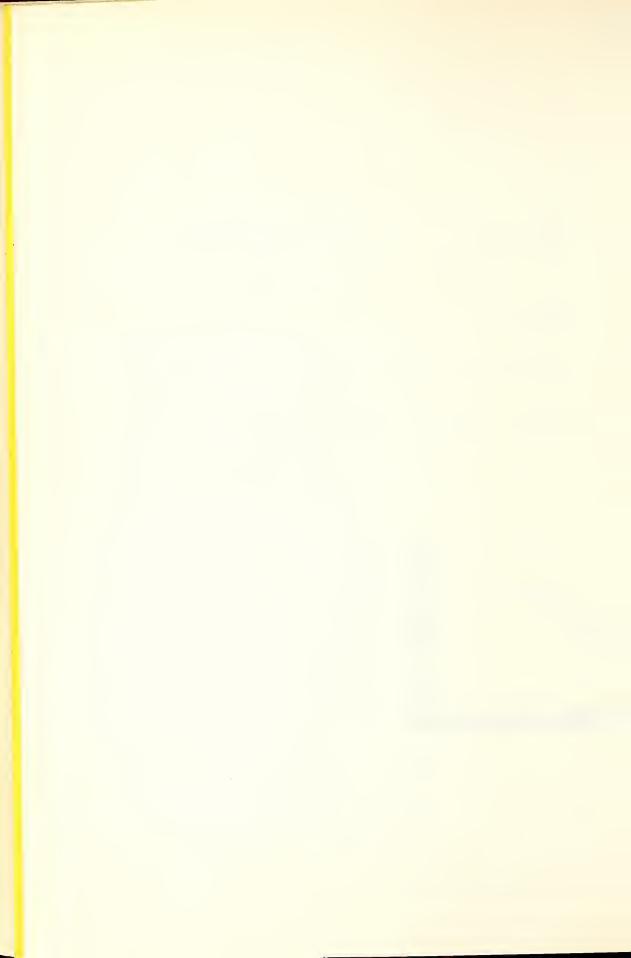
Evaluation of Methoxychlor for the Protection of Stored Wheat and Shelled Corn from Insect Attack



Marketing Research Division

Agricultural Marketing Service

U. S. DEPARTMENT OF AGRICULTURE



#### STATUS OF TOLERANCES FOR METHOXYCHLOR RESIDUES ON STORED WHEAT AND SHELLED CORN

At the present time the tolerance for methoxychlor residues on stored wheat and shelled corn is 2 p.p.m. This level of tolerance was established to cover any residues that might result from spraying empty grain bins with a residual type methoxychlor spray.

The reader is cautioned that no tolerance has been established as yet at levels to permit the use of methoxychlor dusts as discussed in this publication. Until such tolerance is announced, methoxychlor protective treatments should not be used.

This report is the first of a series presenting results of tests with various insecticidal dusts and sprays applied to stored grain for protection against insect attack. These reports are a part of a broad program of research to reduce the cost of marketing farm products, including the cost of preventing insect infestation in stored grain.

February 1958

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#### SIMMARY

This report presents results of tests with various insecticidal dusts and sprays applied to stored grain for protection against insect attack. It summarizes the tests with methoxychlor made in the period August 1953 through December 1956. Because of the interest in protective dusts and sprays for stored grain, results to date are published even though the studies are being continued.

The studies were conducted on wheat and shelled corn stored in the standard circular 3,250-bushel metal bins of the Commodity Credit Corporation, U. S. Department of Agriculture. After the protective treatments were applied the grain was sampled monthly to determine the levels of insect population and moisture content, and quarterly for chemical residue analysis, bioassay tests, and commercial grade. In tests with wheat, the level of residue was also traced through the milling and baking processes.

Dusts applied to wheat in September and October 1955 at rates to give deposits of 5, 10, and 15 p.p.m. of methoxychlor practically eliminated the insect populations present, and kept the wheat almost insect-free through the following season until December 1956. However, when the results were tested for significance by the method of analysis of variance, the difference between the treated and check bins was not significant. The lack of significance was attributed to too short a period of comparison, and the decision was made to continue the observations of these bins until the treatments ceased to give protection or significant differences were obtained.

The amount of methoxychlor recovered from the wheat in December 1955 was 80, 64, and 15 percent, respectively, of the amount applied to the 5, 10, and 15 p.r.m. treatments. In October 1956, at the end of the first year, 40, 45, and 35 percent respectively remained.

The residues on other lots of wheat when received at the mill were less than one-half of the applied amount of 13.6 p.p.m. By the time the wheat had passed through the cleaning and tempering processes, the residues had been reduced to between 1.47 and 2.09 p.p.m. The largest portion of this remainder occurred in the feed fractions (bran and shorts) with small amounts in the second and first clear flours, and less than 0.5 p.p.m. in the patent flour.

The baking process did not destroy residues of methoxychlor but it reduced them by 21 to 44 percent.

There was little change in the overall moisture content of the wheat, and there was no apparent effect on the behavior of the protective dusts. There were no changes in the commercial grade of the wheat during the period of the test.

Dusts applied to shelled corn in August 1953 at rates to give deposits of 54 and 108 p.p.m. gave excellent protection through 1954 and 1955. The differences between the treated lots and checks were highly significant. These differences demonstrated that dosage levels of 54 p.p.m. and above were greater than needed for effective protection over the 30-month observation period.

Dusts applied to shelled corn in August 1954 at rates to give deposits of 36 and 54 p.p.m. gave excellent protection through the following year and until February 1956. The differences between the treated lots and checks were significant. These differences demonstrated that dosage levels of 36 p.p.m. and above were greater than needed for effective protection over the 19-month observation period.

Dusts applied to other lots of shelled corn in August 1954, in which the concentration of methoxychlor was varied but the amount was constant at 45 p.p.m., gave uniformly excellent protection against infestation through the following year and until February 1956. The differences between the treated lots of grain and the checks were not significant. The lack of significance was attributed to too short a period of comparison. It was necessary to terminate the tests so no further observations were made.

When test insects were confined on samples from the treated bins at quarterly intervals, the mortality produced gradually declined from quarter to quarter at about the same rate that the amount of residue, as indicated by chemical analysis, declined.

Analysis of the residues from 16 points in 1 bin of shelled corn revealed a high concentration in the center of the bin beneath the spout of the conveyor.

The moisture content of the samples indicated a continual transfer of moisture within the grain mass, but little change in the average for the bin. There was no apparent effect on the behavior of the protective treatment. There was an effect on the commercial grade in that 25 of the 43 bins under observation were downgraded from 1 to 5 grades because of increases in the amount of total damage which were attributed to moisture accumulation in the surface layer of corn. No downgrading was attributed to the protective treatments.

Five species of stored-grain insects were found in the wheat, the most abundant being the flat grain beetle. However, in corn 17 species were found. The rice weevil was the most abundant species in the bins receiving protective treatments but was insignificant in the check bins, a situation that could not be explained.

## EVALUATION OF MATHOXYCHLOR FOR THE PROTECTION OF STORED WHEAT AND SHELLED CORN FROM INSECT ATTACK

By H. H. Walkden and Howard D. Nelson Stored-Grain Insects Laboratory Manhattan, Kans. 1/

#### INTRODUCTION

Changes in grain handling and storage practices in the past few years have lengthened the periods of time grain may be stored, sometimes up to 5 years. The reserves of grain accumulated as a result of increased yields and the price-support loan program have required new and improved methods of insect control. Emphasis now is being placed on preventive rather than curative measures of preserving grain from insect attack.

In line with this trend, intensive studies were initiated in the fall of 1952 to explore the possibility of applying insecticides directly to stored wheat and shelled corn to prevent the development of insect infestation. The use of insecticides for this purpose introduces numerous problems. The residues of insecticide on the grain must be studied to determine whether they are within safe limits for the ultimate consumer—human beings or livestock—and whether the grade of the grain is affected by the treatments.

This report is the first of a series presenting the results of the tests with various protective treatments. In the tests described here, methoxychlor dusts were used as the protective treatment. Other studies with methoxychlor are still underway, but owing to the immediate interest in protective treatments, the results through December 1956 are presented now. The data are divided into two groups for presentation, one group covering the tests on protection of wheat, the other on shelled corn.

#### TECHNIQUE

The tests were conducted in USDA circular metal bins, 16 feet in height and 18 feet in diameter, with a capacity of 3,250 bushels. The dust applications were made either by hand as the grain was run from the auger, or by a mechanical applicator attached to the tube of the auger.

<sup>1/</sup> This laboratory is a field station of the Stored-Product Insects Section, Biological Sciences Branch, Marketing Research Division, Agricultural Marketing Service, U. S. Department of Agriculture.

Three series of tests were made, 1 with wheat and 2 with shelled corn. The tests with wheat were begun in September 1955, at a CCC bin site in Morris County, Kans., and involved 24 bins. The first series with shelled corn was begun in August 1953 at a CCC bin site in Saline County, Mo., and involved 19 bins. The second series with shelled corn was begun a year later in August 1954, also in Saline County, and it involved 28 bins.

## Sampling Methods

Samples were drawn monthly from 8 places in the grain mass for determinations of the insect population and moisture content of the grain. The samples were drawn with a 5-foot, or 10- or 11-celled, trier equipped with extension handles. Each sample amounted to about 500 grams of grain. The samples were taken vertically in the center of the bin from the top, middle, and bottom 5 feet, from the top 5 feet in each quadrant at a location 3 feet from the wall, and horizontally 2 to 3 inches from the top at the center. Each sample was taken to the laboratory, screened to remove any insects, and the number and species of insects recorded. The moisture content of each sample was then determined with a dielectric type moisture meter. When a bin of grain reached a level of insect population which would cause it to be designated as "weevily" under U. S. grain standards, it was dropped from the series and fumigated.

The sampling pattern in April, July, October, and December was extended to include vertical samples from the middle and bottom 5 feet in each quadrant. These were composited with the other samples from each bin after the insect and moisture records were taken. The composited sample was then separated with a grain divider into two or more subsamples in accordance with the sampling schedule. One subsample was analyzed for methoxychlor residue. A second subsample was cut from the composite sample from each bin at the beginning of the test and annually thereafter or at the termination of the test to establish commercial grade. A third subsample was used in the bioassay tests.

## Statistical Analyses

The performance of the insecticide was evaluated by comparing the treated lots with the untreated controls and with each other on the basis of the number of "bin-months" of protection. The number of "bin-months" of protection was computed by dividing the total number of months of protection for a series of bins having the same treatment by the number of bins in that series. The treatment was considered to give protection until the insect population reached the level at which it would be designated as "weevily"

under the provisions of the U. S. Grain Standards. The grain was designated as "weevily" if a 1,000-gram sample contained living stored-grain insects as follows: Wheat: 2 or more weevils, or 1 weevil and 3 or more bran beetles, or 5 or more bran beetles. Corn: 2 or more weevils, or 1 weevil and 5 or more bran beetles, or 25 or more bran beetles.

The data thus obtained were tested statistically for reliability by the analysis of variance method. This method of statistical analysis establishes the significance of mean differences between treated lots or between treated lots and the untreated controls.

## Chemical Analyses

The analyses of the grain samples for residues of methoxychlor and the lyses of bread were done by a modified Claborn and Beckman method. 2/

The samples from the milling tests were analyzed by the Fairing and Warrington method. 3/

#### TESTS WITH WHEAT

The wheat used in this work was of the 1952 crop. It was delivered from farm storage and placed in CCC storage in the spring of 1953.

These studies were made to determine the degree of protection afforded by the methoxychlor treatments, and the level of insecticidal residues in the wheat, in the milling fractions, and in the bread.

To attain these objectives three types of tests were conducted:

1. Wheat was treated with methoxychlor, and the degree of protection determined by observation of the insect population trends; the amount of residue in the wheat and its persistence during the storage period was determined by chemical analyses of samples drawn periodically from the bins. 2. Wheat with a known amount of residue was milled and the amount of methoxychlor in the screenings, aspirations, and milling fractions was determined by chemical analyses. 3. Bread was baked from flour containing known amounts of methoxychlor and the residue was determined by chemical analyses.

<sup>2/</sup> Claborn, H. V., and Beckman, H. F. Determination of 1,1,1-trichloro-2,2-bis(p-methoxyphenyl)ethane in milk and fatty materials. Anal. Chem. 24: 220-222. 1952.

<sup>3/</sup> Fairing, J. D., and Warrington, H. P. Colorimetric determination of small quantities of 1,1,1-trichloro-2,2-bis(p-methoxphenyl)-ethane. Adv. Chem. Ser. No. 1: 260. 1950.

#### Experimental Pattern

#### Performance Tests

Methoxychlor dusts were applied in September and October 1955 to wheat stored at the Morris County, Kans., bin site. Five bins were treated at the rate of 5 p.p.m. of methoxychlor, 5 bins at 10 p.p.m., and 5 at 15 p.p.m. Nine untreated bins were used as checks. The dusts were formulated with concentrations of 1, 2, and 3 percent methoxychlor and were applied at the rate of 30 pounds per 1,000 bushels of wheat. Wheat flour was used as a carrier since previous experience had shown that an inorganic carrier would impart a gritty feel to wheat and cause it to be downgraded. The wheat was screened as it was turned from one bin to another in order to apply the dusts (fig. 1). The dust was applied with a mechanical applicator attached to the auger tube. The untreated controls were also turned and screened. Each bin was sampled monthly beginning in December 1955 through December 1956, to determine the insect population trends and the moisture content of the wheat. Quarterly and terminal samples for residue analyses and commercial grade were taken as described previously.

#### Milling Tests

Approximately 120 bushels of wheat were treated uniformly with 1 percent of methoxychlor formulated on wheat flour at an application rate of 13.6 p.p.m. The treated wheat was divided into 4 lots of 30 bushels each, 2 of which were milled in September 1954 at the Kansas State College pilot mill and the other 2 at the commercial pilot mill at Minneapolis, Minn. The daily capacity of the mills was: College mill 180 cwt.; commercial mill 105 cwt. Two cleaning processes were employed at each mill, designated as the minimum and maximum. These differed in that the maximum cleaning involved washing the wheat prior to tempering. In addition a 30-bushel lot of untreated wheat was milled at each mill to serve as standards.

During the milling process, samples for determination of methoxychlor residues were taken from 24 points as follows: 8 from the wheat before and during cleaning and tempering process; 6 from different types of screenings; 5 from the different aspirators; 5 from the finals--bran, shorts, second clear flour, first clear flour, and patent flour.

At the Kansas State College mill, all of the screenings, aspirations, and milling fractions were collected and weighed to establish the proportions of each.



Neg. 5182 (B)

Figure 1.--Above, Method of turning grain from one bin to another using two augers and a cleaning screen. At right, Close-up of simple mechanical dust applicator.



Neg. 5182 (C)

#### Baking Tests

Bread was baked from flour to which methoxychlor had been added at rates of 3.75, 7.5, 15.0, and 30.0 p.p.m. Two bakings were made 1 week apart during the winter of 1953-54, and 2 loaves with each concentration were prepared at each baking. Samples from each loaf were analyzed for methoxychlor content to determine the degree to which residues were reduced by the baking process.

## Insect Population Trends

The insect populations in the treated and check bins are shown in table 1. It will be noted that at all 3 levels of treatment the insect populations practically were eliminated following treatment, and the bins remained practically insect free during the 1956 season. It should be recognized that a part of the free-living insect population present before treatment was removed when the wheat was turned and screened. The insect populations in 3 bins of untreated controls increased to the "weevily" level, 1 by March 1956, 1 by August, and 1 by October. At this stage they were fumigated.

### Statistical Analyses of Performance

As previously stated, the criterion for elimination of a bin of grain from a series was whether the insect population had reached a level that would be "weevily" according to United States grain standards. The mean number of months of protection for a series of bins was designated as the number of "bin-months" of protection. The bin-months of protection for the treated series compared with those of the controls are given below:

| Treatment  | Bin-months of protection    |
|--|-----------------------------|
| No treatment - controls 5 p.p.m. 10 p.p.m. 15 p.p.m. | 8.3<br>12.2<br>13.0<br>13.0 |

The differences between the treated lots and the controls or between treated lots were tested for significance by the method of analysis of variance. The analyses showed that the differences were not significant. The variation ratio "F" of 2.24 was less than the significant "F" value for this series of 3.10 at the 5-percent level.

Table 1.--Insect populations in wheat to which specified rates of methoxychlor had been applied in September and October 1955

|  |                              |  |         |   | Livi  | ng insects   | s found pe                             | Living insects found per 1,000 grams of wheat | ams of whe                | at                                      |           |   |   |   |
|--|------------------------------|--|---------|---|---|--|--|---|---------------------------|---|-----------|---|---|---|
| Application rate and test number   | Refore                       | 1955                                     |         |   |   |  |  | 1   | 1956                      |   |           |   |   |   |
|  | treatment                    |  | January | February  | March   | April  | May                                    | June  | July                      | August                                  | September | October                                       | November  | December                                  |
|  | Number                       | Number                                   | Number  | Munber  | Number  | Number   | Mumber                                 | Number  | Mumber                    | Number                                  | Number    | Number  | Number  | Number                                    |
| 5 p.p.m. (30 lbs./1,000 bu. of l-percent concentration)  1 2 2 3 4 4 5 5 | 4.12<br>. 88<br>. 76<br>1.12 | 0000                                     | 11111   | 0000  | 00000   | 00000  | 00000                                  | 00000   | 00000                     | 00000                                   | 00.1/1.0  | 0.1<br>.25.<br>.25.                           | 000 0   | 000 0                                     |
| 10 p.p.m. (30 lbs./l,000 bu. of 2-percent concentration) 6. 7. 8 9 9     |                              | 00000                                    | 11111   | 00000   | 00000   | 00000  | 00000                                  | 00000   |                           | 00000                                   | 00000     | . 2. 0<br>0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 | 00000   | 00000                                     |
| 15 p.p.m. (30 lts./l,000 bu. of 3-percent concentration) 11 12 13 14 15  | 0<br>0.04<br>1.70<br>.82     | 00000                                    |         | 00000   | 00000   | 00000  | 00000                                  | 00000   | 00000                     | 00000                                   | 00000     | 00000   | 00000   | 00000                                     |
| No application - controls 16 17 18 20 21 22 23 23 23                     | No<br>counts                 | 13.0<br>2.5<br>0.0<br>0.5<br>1.5<br>1.25 |         | 2/7.75<br>0<br>0<br>0<br>0<br>0<br>2/8.25<br>1.75 | 2/5.0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0 | 77.<br>0 0 0 0 0 1 1 0 0 0 1 2 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 | 20000000000000000000000000000000000000 | 0.25  | , 0, 000 , 0 w<br>N N N N | 2 2 1 1 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 |           | 2/<br>2/2/2/2/2/2/2/2/2/2/2/2/2/2/2/2/2/2/    | 11.00<br>3.00<br>3.00<br>3.00<br>3.00<br>3.00<br>3.00<br>3.00 | 25.<br>11.25<br>12.25<br>0 0 57.<br>0 75. |
|  |                              |  |         |   |   |  |  |   |                           |   |           |   |   |   |

1/ Test torminated; grain reached weevily stage.  $\overline{2}/$  After this count the tin was funigated.

The lack of significance, even though the treated bins were kept practically insect free throughout the period of the tests, was due to the fact that insect infestation did not develop rapidly in the check bins. Therefore, a longer period of comparison was needed. The decision was made to continue the observations of these bins until the treatments ceased to give protection or significant differences are obtained.

#### Insecticidal Residues

#### In the Wheat

The residues found in the composite samples (prepared by combining samples from 16 locations) at quarterly intervals are given in table 2.

The amount of methoxychlor recovered in the first analysis in December 1955 was 80, 64, and 45 percent, respectively, from the 5, 10, and 15 p.p.m. applications. In October 1956, at the end of the first year, 40, 45, and 35 percent respectively of the 5, 10, and 15 p.p.m. applications remained.

In the Screenings, Aspirations, and Milling Fractions

The residues found in the milling products are given in table 3. The results showed that about half of the freshly applied methoxychlor was recovered from the wheat as received for milling. By the time the wheat had passed through the cleaning and tempering processes the residues had been reduced to a range of 1.47 to 2.09 p.p.m. The largest portion of the residues were found in the feed fractions (bran and shorts), with small amounts in the second and first clear flour, and less than 0.5 p.p.m. in the patent flour.

#### In the Bread

The residues found in bread made from flour to which methoxychlor had been added are given in table 4. These results demonstrated that baking did not destroy residues of methoxychlor, although the amounts were reduced 21 to 44 percent as shown in table 5.

Table 2. -- Residues on wheat at various intervals during storage following applications of methoxychlor at 5, 10, and 15 p.p.m. in September and October 1955

| eat                    | October<br>1956  | P.p.m. |  |  |
|------------------------|------------------|--------|--|--|
| <br>oxychlor on wheat  | July<br>1956     | P.p.m. |  |  |
| Amount of methoxychlor | April<br>1956    | P.p.m. |  |  |
|                        | December<br>1955 | P.p.m. | SOLIOU TOWNTER OCHOOCO TOWNTER   |  |
| Application rate and   | test number      |        | 5 p.p.m. (30 lbs./l,000 bu. of  1-percent concentration)  2.  3.  4.  5.  Mean  10 p.p.m. (30 lbs./l,000 bu. of  7.  8.  9.  10.  Mean  15 p.p.m. (30 lbs./l,000 bu. of  12.  13.  14.  15 p.p.m. (30 lbs./l,000 bu. of  11.  12.  13.  14.  15. |  |

Table 3.--Residues in various milling products from wheat treated with methoxychlor dust at the rate of 13.6 p.p.m.; treated and milled in September 1954

| Before and during cleaning and tempering Wheat as received at mill Wheat after Carter disc Wheat after aspirator Wheat after washer. Wheat after tempered wheat scourer Wheat after Entoleter scourer aspirator From Millerator From Garter disc. From Aspirator From aspirator. From aspirator. From aspirator. From day scourer aspirator. From tempered wheat scourer From Entoleter scourer aspirator. From Entoleter scourer aspirator. From Millerator. From Millerator. From Aspirator. From Entoleter scourer aspirator. From Aspirations From Aspirator. From Entoleter scourer aspirator. From Entoleter scourer aspirator. From Entoleter scourer aspirator. From Entoleter scourer aspirator. | Perportion of original weight of wheat Percent 100,000 100,000 772 222 207 222 054 054 057 222 054 057 2093 222 093 093 093 093 093 093 093 093 093 093 | Milled at Manhattan Cleaning series Series series P.p.m. P.p.m. 5.97 5.32 2.19 2.60 2.19 2.38 2.38 2.38 2.38 2.38 2.38 2.38 2.38 | Maximum cleaning series P.D.m. 3.27 2.41 2.30 1.47 2.41 2.30 1.47 2.41 2.80 1.20 5.41 | Minimum cleaning series series series series series series series series series 3.09 2.80 3.01 2.51 3.15 3.15 3.15 2.09 1.95 2.09 1.95 1.95 1.95 1.95 1.95 1.95 1.95 1.9 | Inneapolis  Maximum cleaning series P.p.m. 2.80 2.51 3.41 2.87 3.18 2.33 1.95 4.96 42.02 13.68 |
|---|---|--|---|--|--|
| Finals<br>Bran  | 21,602  | 6.39<br>3.25   | 4.62<br>3.18  | 6.65<br>1/6.28<br>2/1.26   | 6.67<br>1/5.29<br>2/3.55   |
| 2nd clear flour   | 3.656<br>10.126<br>53.956<br>97.746   | 2.16<br>.52<br>.14   | 2.62<br>1.07<br>.38   | 2.01<br>1.26<br>.55  | 3.34<br>1.71<br>1.49   |

 $\frac{1}{2}$  Head end shorts.

Table 4.--Residues in bread baked from flour containing known amounts of methoxychlor,
November 1953 - March 1954

| Concentration of         | Amount                       | of metho                     | xychlor p                    | present i                    | n bread fi                   | rom                          |                              |
|--------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|
| methoxychlor             | Fi                           | irst bakin                   | ng                           | Sec                          | cond bakil                   | ng                           | 4-loaf<br>mean               |
| in flour <u>l</u> /      | Loaf<br>No.1                 | Loaf<br>No.2                 | Mean                         | Loaf<br>No.1                 | Loaf<br>No.2                 | Mean                         |                              |
|                          | P.p.m.                       |
| P.p.m 3.75 7.5 15.0 30.0 | 1.53<br>2.86<br>4.92<br>7.60 | 1.48<br>2.34<br>5.00<br>8.20 | 1.51<br>2.60<br>4.96<br>7.90 | 1.28<br>2.70<br>5.48<br>9.76 | 1.30<br>2.82<br>5.40<br>9.76 | 1.29<br>2.79<br>5.44<br>9.76 | 1.40<br>2.68<br>5.20<br>8.83 |

<sup>1/</sup> The amounts found by chemical analysis were respectively: 2.73, 6.28, 13.40, and 24.32 p.p.m.

Table 5.--Reduction of residues during the baking process of bread made from flour containing known amounts of methoxychlor

| Amount of methoxychlor | Mean                     | weight                           | Mean amount of methoxychlor  | Reduction of residuel/ |
|------------------------|--------------------------|----------------------------------|------------------------------|------------------------|
| in flour               | Flour                    | Loaves                           | in bread                     | residuel               |
| P.p.m                  | Grams                    | Grams                            | P.p.m.                       | Percent                |
| 2.73                   | 315<br>315<br>315<br>315 | 484.5<br>486.8<br>488.0<br>487.8 | 1.40<br>2.68<br>5.20<br>8.83 | 14<br>140<br>140<br>21 |

<sup>1/</sup> Reduction of residue=(Weight of flour (Weight of loaf x residue in flour) - residue in bread x100

Weight of flour Weight of loaf x residue in flour

### Moisture Content Changes in the Wheat

The wheat used in these tests was of low moisture content, most of it being within the range of 10.5 to 11.5 percent. The moisture content of the samples taken from 16 locations in the bin showed that there was a continual transfer of moisture within the grain mass, but there was little change in the overall content. There was no apparent effect on the behavior of the protective treatments attributable to variations in moisture content, nor was there any evidence that the protective treatments influenced the transfer of moisture or the amount of the moisture content.

## Changes in the Commercial Grade

There were no changes in the commercial grade of any bin during the period of the test.

#### TESTS WITH SHELLED CORN

The tests with shelled corn were directed toward determining the degree of protection against insect infestation afforded by the methoxychlor treatments, the level of insecticidal residues, the distribution of the insecticide, changes in the moisture content, and changes in commercial grade during the storage period.

## Experimental Pattern

In the first series of tests, the dust was applied in August 1953. Six bins were treated with 10 percent methoxychlor in corncob flour at the rate of 5h p.p.m. of methoxychlor, and 6 at the rate of 108 p.p.m. In the first group the dust formulation was applied at the rate of 30 pounds per 1,000 bushels and in the second group at 60 pounds per 1,000 bushels. The corn was shelled on the farm and delivered directly to the bin site. As the corn from each truck was emptied into the hopper of the auger, aliquots of the dust were applied by hand. The dust tended to be sticky and did not flow well, but it was expected to become well distributed as the corn passed through the auger and cascaded into the bins.

In the second series, started in August 1954, treatment was delayed until all the corn had been loaded into the bins, and then the dust was applied as the corn was turned from one bin to another. The dust was applied by hand in aliquots as the grain dropped from the spout of the unloading auger to the hopper of the elevating auger. Two groups of tests were made, one to compare

two application rates of a selected concentration. Five bins each were treated with a 5-percent concentration at amounts to give 36 and 54 p.p.m. of methoxychlor. The other group of tests was to study the effect of varying the concentrations of methoxychlor in the protectant material while holding the total amount constant. Three bins each were treated with 2.5, 3.33, 5, and 10 percent concentration of methoxychlor in corncob flour at amounts to give an application rate of 45 p.p.m. of methoxychlor. Six untreated bins served as controls.

Each bin in both groups was sampled at monthly intervals following treatment until January or February 1956 to determine insect population trends and moisture content. A 5-foot grain trier or probe was used as in the wheat. The same pattern of sampling was used as for wheat, consisting of 8 probings, 1 in the top 5 feet in each quadrant, in the top, middle, and bottom 5 feet in the center, and 1 horizontally in the surface layer. This pattern was extended at each quarter to 16 probe samples consisting of the top, middle, and bottom 5 feet in each quadrant and the center, and 1 horizontal in the surface grain. After the insect population and moisture content of the corn had been determined, these samples were composited and samples were cut with a grain divider for residue analyses, commercial grade, and bioassay tests.

Bioassay tests were conducted for the purpose of observing any changes in the potency of the insecticide during the storage period. In these tests, 25 adults each of rice weevils and flour beetles were confined on 3 ounces of corn from both the treated and untreated bins and the resulting mortality recorded after 1 and 3 weeks.

In 1 bin all 16 of the samples were analyzed separately to obtain information on the distribution pattern of the insecticide.

## Insect Population Trends

The trends of the insect populations in both the treated and control bins of the series started in August 1953 are presented in table 6. In general the insect populations remained very low throughout the remainder of the 1953 season, as well as through 1954 and 1955. One bin treated with 54 p.p.m. became weevily by November 1954. At 108 p.p.m., one bin reached this stage of infestation in the winter of 1954-55, and a second bin by July 1955. All of the controls for the 1953 season developed large populations by September or October; for the 1954 season by summer or fall; and for the 1955 season by August or September.

Table 6.--Insect populations in shelled corn following applications of methoxychlor at 54 and 108 p.p.m. in August 1953

|                                | 1956                             | Jan.                      | No.        | 1.8   | 0000  | 199   |
|--------------------------------|----------------------------------|---------------------------|------------|---|---|---|
|                                |                                  | Dec.                      | No.        | 0.8   | 1.0<br>0  | ψ.ν.<br>ν.ν.  |
|                                |                                  | Nov.                      | No.        | 11 17   | 111 1   | 1.1   |
|                                |                                  |                           | No         | 0 .<br>1 . شقق  | 0000  | 3.8   |
|                                |                                  | Sept. Oct.                | No.        | 0.1   | 0 0 0   | 28.0<br>125.8<br>8.8  |
|                                |                                  | Aug.                      | No.        | 0   | ه المان   | 16.0<br>14.3<br>6.8   |
|                                | 1955                             | July Aug.                 | 8          | 00 0 0  | 0.00  | 9.000<br>7.   |
|                                |                                  | June                      | %<br> <br> | 00 00   | 00000   | 4.5<br>0<br>0   |
|                                |                                  | May                       | s<br>S     | 00 0 0  | 00000   |   |
|                                |                                  | Apr. May                  | No.        | 00 000  | ,0000   | 0000  |
| corn                           |                                  | Mar.                      | No.        | 00 000  | ۵,0000  | 000   |
| e11ed                          |                                  | Feb.                      | No.        | 00 0 00   | ,000°,0   |   |
| of shelled corn                |                                  | Jan.                      | No.        | 00 000  | 00.00   |   |
| grams                          |                                  | Dec.                      | N<br>08    | 0000  | NOO 0   | 77  |
|                                |                                  |                           | No         | 000000000000000000000000000000000000000   |   | 20°2<br>8   |
| er l,                          |                                  | Sept. Oct. Nov.           | No         | 0<br>0,0<br>0,5<br>0,5  | ,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,  | ي.<br>بر  |
| d pun                          |                                  | Sept.                     | No         | 000 0   | *0000 <b>0</b>  | 8<br>8  |
| Living insects found per 1,000 |                                  | Aug.                      | No.        | 0 0   | 000 00  | 8.8<br>Y  |
| insec                          | 1954                             | July Aug.                 | No         | 000 000   | ,<br>00000  | 0 13.5  |
| ving                           |                                  | June                      | No         | 000000  | 00 000  | £, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1, 1,   |
| E                              |                                  | May                       | No.        | 000000  | 0,0000  | £ 7.  |
|                                |                                  | Apr.                      | 9          | 000000  | 000000  | w r   |
|                                |                                  | Mar.                      | ့<br>မွ    | 000000  | 0000 0  | 0 0   |
|                                |                                  | Feb.                      | No.        | 000000  | 0000.0  | rů ů  |
|                                |                                  | Jan.                      | No.        | 111111  | 11111   | 1 1   |
|                                |                                  | Dec.                      | No.        | 0440  | , 0000<br>mm  | 1.0   |
|                                |                                  | Nov.                      | No.        | 0   | 0   | 71.5.5.3.1<br>11.1.2.8.8.8.1  |
|                                | 1953                             | Oct.                      | No.        | 0   | 2<br>   | 37.5<br>8.0<br>17.5<br>8.9<br>19.8<br>19.8  |
|                                |                                  | Aug. Sept. Oct. Nov. Dec. | No         | 00 00 ° 1   | 8 0 0 E   | 8<br>8 8 8 8 9 9 9 9 9 9 9 9 9 9 9 9 9 9 9  |
|                                |                                  | Aug.                      | No.        | 0000 - 1  | 00000 m   | 0 8.3 37.5<br>0 8.0 17.5<br>0 8.0 17.5<br>2.0 12.0 14.5<br>1.8 114.3 92.8<br>1.3 10.8 32.3<br>1.3 10.8 32.3 |
|                                |                                  |                           |            | ų · · · · · ·   | 9 · · · · · ·   |   |
|                                | and                              |                           |            | bu.   | on)   |   |
|                                | rate                             |                           |            | 1,000<br>trati  | 1,000   | ontro   |
|                                | Application rate and test number |                           |            | lbs./   | oncer.  | ō   |
|                                | tes                              |                           |            | (30   | ent c   | ation   |
|                                | Ap                               |                           |            | 54 p.p.m. (30 lbs./1,000 bu. of 10—percent concentration) 1. 2. 3. 3. 4. 6. 6. 6. 6. 6. 6. 6. 6. 6. 6. 6. 6. 6. | 100 p.p.m. (60 lbe,/l,000 bu. of lopercent concentration) 7. 8. 8. 8. 8. 8. 8. 8. 8. 8. 8. 8. 8. 8. | No application - controls 13  |
|                                |                                  |                           |            | 54 p  | 108   | N <sub>O</sub>  |

1/ Test terminated; grain reached weevily stage.

The insect populations in the series started in August 1954 are presented in table 7. An application rate of 36 p.p.m. gave excellent protection through the remainder of 1954 and all of 1955 as compared with the check bins, except in one bin that developed some infestation in October 1955. The rate of 54 p.p.m. gave excellent protection throughout. Varying the amount of diluent with a constant amount of methoxychlor equal to 45 p.p.m. did not vary the effectiveness of the protection since all applications gave excellent results except one bin which became "weevily" in September 1955. All controls began to develop infestation during the fall of 1954, and were seriously infested by September 1955.

## Statistical Analyses of Performance

The data on the performance of the insecticide as applied to corn were handled in the same manner as for wheat. Bins were eliminated from a series if they became weevily, and the number of bin-months of protection was computed as for wheat.

The bin-months of protection for the series of tests begun in August 1953 are given below:

| Treatment                                    | Bin-months of protection |
|--|--------------------------|
| No treatment - controls 54 p.p.m. 108 p.p.m. | 5.3<br>26.2<br>26.8      |

When tested for significance by the method of the analysis of variance, the mean difference between the treated series was not significantly different, but the difference between the lots treated and the untreated controls was highly significant. The variation ratio "F" was 33.38, a much greater value than the 3.63 required for significance for this group.

The highly significant difference between the treated and the control lots demonstrated that dosage levels of 5h p.p.m. and higher were greater than needed for effective protection over the 30-month observation period.

The bin-months of protection for the first group of the tests begun in August 1954 are given below:

| Treatment               | Bin-months of protection |
|-------------------------|--------------------------|
| No treatment - controls | 15.8                     |
| 36 p.p.m.               | 19.0                     |
| 54 p.p.m.               | 19.0                     |

Table 7 .-- Insect populations in shelled corn following applications of methoxychlor at rates of 36 and 54 p.p.m., and at a constant rate of 45 p.p.m., in August 1954

|   | L        |   |   |         |               |                  | Living       | insects | per    | 1,000 gr | grams of | shelled                                 | corn                 |                     |                     |        |        |        |                 |
|---|----------|---|---|---------|---------------|------------------|--------------|---------|--------|----------|----------|---|----------------------|---------------------|---------------------|--------|--------|--------|-----------------|
| Application rate and test number  |          |   | 1954                                      |         |               |                  |              |         |        |          | 1955     | 55                                      |                      |                     |                     |        |        | 15     | 1956            |
|   | Aug.     | Sept.                                   | Oct.                                      | Nov.    | Dec.          | Jan.             | Feb.         | Mar.    | Apr.   | May      | June     | July                                    | Aug.                 | Sept.               | Oct.                | Nov.   | Dec.   | Jan.   | Feb.            |
|   | Number   | Number                                  | Number                                    | Mumber  | Number        | Number           | Number       | Number  | Number | Number   | Number   | Number                                  | Number               | Number              | Number              | Number | Number | Number | Number          |
| 36 p.p.m. (40 lbs./1,000 bu. of 5-percent concentration) 1 2 3 4 4 5    | 2.77.00  | 2.25                                    | 0.25                                      | 0000    | 00000         | 00000            | 00000        | 00000   | 00000  | 00000    | 00000    | 0 | 0 0 1.25             | 00000               | 0<br>.25<br>.0<br>0 | 11111  | 0000   | 11111  | 3.25            |
| 54, p.p.m. (60 lbs./1,000 hu. of Spercent concentration) 6 7 8 9 10     | 8,00 v y | 0000 W.R.                               | 00<br>~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~ | 00.00   | 00000         | 00000            | 00000        | 00000   | 00000  | 00000    | 00.00    | 00000                                   | 00000                | 1.25<br>0<br>0<br>0 | 0000                | 11111  | 00000  | 11111  | 0 0 0 25        |
| 45 p.p.m. (25 lbs./l,000 bu. of lo-percent concentration) 11            | , , , o  | .25<br>1.75                             | 000                                       | 0.25    | 000           | 0 0              | 000          | 000     | 000    | 000      | 000      | 000                                     | 0.75                 | 000                 | 000                 | 111    | 000    | 111    | 000             |
| h5 p.p.m. (50 lbs./1,000 bu. of 5-percent concentration) . 1h . 1h . 15 | 1.50     | , v v v v v v v v v v v v v v v v v v v | 000                                       | 000     | 000           | 000              | 000          | 000     | 000    | 000      | 000      | 0000                                    | 1.50                 | о<br>п,             | 7,00                | 11     | 0 .25  | 11     | 00              |
| h5 p.p.m. (75 lbs./1,000 bu. of 3.33-percent concentration)             | 22.0     | 1.25                                    | 000                                       | 00 0.2% | 000           | 000              | 000          | 000     | 000    | 000      | 000      | 000                                     | 000                  | 000                 | 000                 | 111    | 000    | 111    | 000             |
| 45 p.p.m. (100 lbs./1,000 bu. of 2.5-percent concentration) 20 21 22    | 0,0      | .25                                     | 000                                       | 0.0     | 000           | 000              | 000          | 000     | 000    | 000      | 000      | 000                                     | .25                  | 000                 | 00                  | 111    | 000    | 111    | 000             |
| No application - controls 23 24 24 25                                   | 2.25     | 5.75<br>1.25<br>1.25<br>1.25            | 2.75                                      | 2000    | 22.5          | 3.25             | .75          | 1.0     | 0 0    | 000      | 1.25     | 1.25                                    | 9.75<br>13.25<br>5.0 | 18.5                | 3,50                | 1      | 3.50   | 1      | 1.25            |
| 26  | 2.25     | 4.75<br>4.25<br>4.25                    | 2.25                                      | 1.25    | 0.4.0<br>7.7. | 2.2.5.<br>2.2.5. | ч.<br>o.v.v. | 25.55   | 0.00   | 000      | 000      | 2.75                                    | 6.0                  | 23.50               | 2.25<br>1,0         | 11     | 20.0   | 17     | 4.5<br>-1/127.0 |
| 1/ Test terminated: grain reached weevilv                               | weevil   | v stage                                 |   |         |               |                  |              |         |        |          |          |   |                      |                     |                     |        |        |        |                 |

1/ Test terminated; grain reached weevily stage.

When tested for significance by the method of the analysis of variance, the differences between the treated series were not significantly different, but the differences between the treated lots and the untreated controls were significant. The variation ratio "F" of 5.76 was greater than the value of 3.68 required for significance for this series.

The significant differences between the treated lots and the controls demonstrate that dosage levels of 36 p.p.m. and above were greater than needed for effective protection over the 19-month period of observation.

The bin months of protection for the second group of the tests begun in August 1954, in which the concentration of methoxychlor in the dust was varied but the total amount held constant at 45 p.p.m. are given below:

| Concentration in dust and dosage | Bin-months of protection |
|----------------------------------|--------------------------|
| No treatment - controls          | 15.8                     |
| 25 pounds of 10 percent dust     | 19.0                     |
| 50 pounds of 5 percent dust      | 17.0                     |
| 75 pounds of 3.33 percent dust   | 19.0                     |
| 100 pounds of 2.5 percent dust   | 19.0                     |

When tested for significance by the method of the analysis of variance, the differences between the treated lots, or between the treated lots and the controls were not significant. The variation ratio "F" of 1.67 was less than the significant value of 3.18.

The situation here parallels that in the wheat tests, in that a longer period of comparison was needed to permit differentiation. It was also necessary to terminate this group of tests so no further observations were made.

## Insecticidal Residues

The residues found in the composited samples taken quarterly are given in tables 8 and 9. Consecutive analyses from a given test were often variable. This may have been due to a combination of uneven distribution of the methoxychlor in the grain (see discussion, p.12) and sampling error.

Two facts were demonstrated by these data, the first that the residue in the corn at the first analysis was in the order of one-half of the application rate; the second that the depletion of the methoxychlor with age was very gradual.

Table 8.--Residues in shelled corn at intervals during storage following applications of methoxychlor at 5μ and 108 p.p.m. in August

|   |                                     | October  | P.p.m. | 19.8<br>15.0<br>20.2<br>18.3   | 23.3<br>145.9<br>147.14<br>38.9 |
|---|-------------------------------------|----------|--------|--|---------------------------------|
|   | 2                                   | July     | P.p.m. | 14.1<br>8.3<br>17.1<br>13.2  | 22.4<br>27.6<br>22.1<br>24.1    |
| in  | 1955                                | April    | P.p.m. | 12.9<br>8.3<br>10.5<br>10.6  | 20.0<br>29.9<br>17.0<br>22.3    |
| Amount of methoxychlor present in shelled corn in |                                     | January  | Р.р.т. | 18<br>112.2<br>116.2<br>8  | 27.3<br>57.0<br>1.3.6<br>1.2.6  |
| sent in sh  |                                     | November | P.p.m. | 18.0<br>12.0<br>28.0<br>19.3   | 29.0<br>86.0<br>61.0<br>58.7    |
| ychlor pre  | 11                                  | July     | P.p.m. | 39.0<br>22.0<br>29.7   | 51.0<br>168.0<br>94.0<br>104.3  |
| of methox   | 1954                                | April    | P.p.m. | 19.0<br>28.0<br>23.0<br>22.7   | 48.0<br>200.0<br>92.0<br>113.3  |
| Amount  |                                     | January  | P.p.m. | 30.0<br>11.0<br>0<br>13.7  | 81.0<br>70.0<br>37.0<br>62.7    |
|   | 3                                   | October  | P.p.m. | 16.0<br>13.0<br>23.0<br>17.3   | 16.0<br>148.0<br>39.0<br>14.3   |
|   | 1953                                | August   | P.p.m. | 23.2<br>31.0<br>25.1   | 50.0<br>63.0<br>59.0<br>57.3    |
|   | Application rate and test number 1/ |          |        | 54 p.p.m. (30 lbs./l,000 bu. of 10-percent concentration) 1 2 4 Mean Mean (60 lbs./l,000 bu. of 100 pu. of 100 | 8                               |

1/ Test numbers correspond with those in table 6.

Table 9.--Residues in shelled corn at intervals during storage following applications of methoxychlor at 36 and 54 p.p.m., and at a constant rate of 45 p.p.m., in August 1954

| Application rate and   | Amount of                                    | methoxychl                                   | or presen                                   | t in shelle                                  | ed corn on                                   |
|--|--|--|---|--|--|
| test number 1  | November                                     | January<br>1955                              | April<br>1955                               | July<br>1955                                 | October<br>1955                              |
|  | P.p.m.                                       | P.p.m.                                       | P.p.m.                                      | P.p.m.                                       | P.p.m.                                       |
| 36 p.p.m. (40 lbs./1,000 bu. of 5-percent concentration)       |  |  |   |  |  |
| 1  | 35.0<br>31.0<br>15.0<br>19.0<br>17.0<br>23.4 | 22.0<br>15.5<br>28.0<br>20.0<br>20.0<br>21.1 | 7.6<br>3.9<br>12.4<br>18.0<br>14.0<br>11.2  | 19.1<br>11.2<br>12.9<br>18.6<br>15.6         | 21.1<br>15.4<br>20.4<br>24.0<br>26.4<br>21.5 |
| 54 p.p.m. (60 lbs./1,000 bu. of 5-percent concentration)       |  |  | ,   |  |  |
| 6  | 39.0<br>39.0<br>21.0<br>21.0<br>22.0<br>28.4 | 13.3<br>27.0<br>37.0<br>28.0<br>42.0<br>29.5 | 9.0<br>18.6<br>10.8<br>11.9<br>19.2<br>13.9 | 22.4<br>20.9<br>21.6<br>14.8<br>16.8<br>19.3 | 26.4<br>30.0<br>36.0<br>33.6<br>28.8<br>31.0 |
| 45 p.p.m. (25 lbs./1,000 bu. of 10-percent concentration) 11   | 35.0<br>36.0<br>22.0<br>31.0                 | 27.0<br>28.0<br>32.0<br>29.0                 | 16.7<br>16.7<br>18.1<br>17.2                | 19.1<br>12.8<br>16.2<br>16.0                 | 24.0<br>37.2<br>28.8<br>30.0                 |
| 45 p.p.m. (50 lbs./1,000 bu. of 5-percent concentration) 14    | 38.0<br>39.0<br>17.0<br>31.3                 | 25.0<br>30.0<br>14.0<br>23.0                 | 11.0<br>15.2<br>20.0<br>15.4                | 14.8<br>18.1<br>16.7<br>16.5                 | 21.0<br>28.0<br>31.2<br>26.7                 |
| 45 p.p.m. (75 lbs./1,000 bu. of 3.33-percent concentration) 17 | 38.0<br>32.0<br>36.0<br>35.3                 | 34.2<br>22.0<br>21.0<br>25.7                 | 13.0<br>16.9<br>7.2<br>12.4                 | 10.5<br>15.7<br>11.2<br>12.5                 | 23.3<br>26.4<br>20.4<br>23.4                 |
| 45 p.p.m. (100 lbs./1,000 bu. of 2.5-percent concentration) 20 | 40.0<br>32.0<br>14.0<br>28.7                 | 19.0<br>19.0<br>35.0<br>24.3                 | 10.2<br>17.1<br>13.8<br>13.7                | 17.1<br>13.3<br>16.2<br>15.5                 | 21.6<br>48.0<br>34.8                         |

<sup>1/</sup> Test numbers correspond with those given in table 7.

## Distribution of the Insecticide

The distribution of methoxychlor in 1 bin at the 16 sampling points is presented in table 10. It was evident that much of the insecticide collected in the center of the bin beneath the spout of the conveyor.

## Bioassay Tests

The results of the bioassay tests are presented in table 11 and corroborate the data obtained in residue analyses (tables 8 and 9) that the depletion of the methoxychlor with age was very gradual.

## Changes in Moisture Content

The corn used in these tests was dry enough to store well, the moisture ranging from 13.4 percent downwards. The moisture determinations of the samples drawn at monthly intervals during the observation period showed that there was a continual transfer of moisture within the grain mass. The subject of moisture transfer has been discussed by Holman et al. and Schmidt 5. In this series of tests there was but little change in the overall moisture content, the usual moisture accumulation being noted in the surface grain during the winter months, followed by a disappearance and redistribution of it during the warmer months of the year. There was no apparent effect on the behavior of the protective treatment.

## Changes in the Commercial Grade

The commercial grades of the different bins of corn, taken at the beginning of the study and at quarterly intervals thereafter, showed that there was no downgrading due to treatment. However, the corn in 25 of the 43 bins under observation was downgraded from 1 to 5 grades because of the increase in the amount of total damage. The increase in the grade factor "Total Damage" during the storage period was due largely to moisture accumulation in the surface grain resulting in varying amounts of spoilage, chiefly in the surface corn in the center of the bin.

L/ Holman, L. E., Barre, H. J., Cotton, R. T., and Walkden, H. H. Storage of Dry Shelled Corn in Farm-type Bins. U. S. Dept. Agr. Cir. 826, pp. 17-19, illus. 1949.

<sup>5/</sup> Schmidt, J. L. Wheat Storage Research at Hutchinson, Kans., and Jamestown, N. Dak. U. S. Dept. Agr. Tech. Bull. 1113, pp. 13-16, 1955.

Table 10.--Distribution of methoxychlor in a 3,250-bushelbin of shelled corn receiving an application in August of 60 lb./1,000 bu. of 10-percent dust (108 p.p.m.), and sampled in October 1953

| Level in grain  | Amount                       | of methox              | ychlor pre            | esent on s          | shelled co           | rn in                           |
|---|------------------------------|------------------------|-----------------------|---------------------|----------------------|---------------------------------|
| rever in grain  | Center of bin                | North<br>quadrant      | East<br>quadrant      | South quadrant      | West<br>quadrant     | Mean                            |
|   | P.p.m.                       | P.p.m.                 | P.p.m.                | P.p.m.              | P.p.m.               | P.p.m.                          |
| Surface Top 5 feet Middle 5 feet . Bottom 5 feet . Mean | 2<br>318<br>495<br>77<br>223 | 13<br>11<br>73<br>32.3 | 8<br>28<br>Ակ<br>26.6 | 9<br>56<br>85<br>50 | 13<br>10<br>58<br>27 | 72<br>120<br>67<br><u>1</u> /87 |

<sup>1/</sup> Mean including surface sample -- 81 p.p.m.

#### COMPARATIVE ABUNDANCE OF THE SPECIES OF INSECTS

The monthly samples drawn from the bins were examined in the laboratory and the number and species of stored-grain insects were recorded. The comparative abundance of the different species is given in tables 12 and 13, expressed as percentages of the total number of insects found during 3-month periods.

In wheat, only five species of stored-grain insects were found, the most abundant species being the flat grain beetle, followed by the saw-toothed grain beetle and the lesser grain borer (table 12).

The rice weevil was the most abundant species found in the series of bins of shelled corn treated with methoxychlor (45 percent of the total insects observed), whereas in the control series rice weevils comprised only a little more than 1 percent of the total. The reasons for this situation are not clear. In the control series, the most abundant species in corn was the red flour beetle, followed by the flat grain beetle and the saw-toothed grain beetle. These 3 species constituted nearly 90 percent of the total insects observed in the controls. A total of 17 species of stored-grain insects were found in corn during the observation period, August 1953 to February 1956 (table 13).

Table 11.--Mortality of adult rice weevils in bicassay tests with samples taken at intervals from the shelled corn treated with methoxychlor protective dusts

| in                                    | 1955                                       | Jamuary April | Percent Percent | 56 54<br>83 82                              | 73 90<br>89 98                               | л<br>1 1                         | 74 79<br>100 95                            | 84 57<br>97 100                            | 74 78<br>97 96                              | 79 62<br>96 92                             | 95 82<br>100 100                              | 58 71<br>90 91                               | 9 1                       |
|---------------------------------------|--|---------------|-----------------|---|--|----------------------------------|--|--|---|--|---|--|---------------------------|
| sampling1/                            |  | October       | Percent         | 71.<br>113                                  | 83   |                                  | 93   | 69   | 62  | 4,9<br>91                                  | 72<br>100                                     | η6<br>9η                                     | 91                        |
| Mortality in bioassay tests following |  | July          | Percent         | 29<br>64                                    | ή8<br>99                                     | 0 0                              | 95   | 95   | 95<br>100                                   | 94<br>100                                  | 98<br>88                                      | 88<br>100                                    | 52                        |
| ssay tests                            | 1954                                       | April         | Percent         | 647   | 9 66   | 0 8                              |  |  |   |  |   |  |                           |
| ty in bioas                           |  | January       | Percent         | 83  | 95   | N M                              |  |  |   |  |   |  |                           |
| Mortali                               | 53   | October       | Percent         | 63  | 46<br>94                                     | 10                               |  |  |   |  |   |  |                           |
|                                       | 1953                                       | July          | Percent         | 89  | 93   | чл                               |  |  |   |  |   |  |                           |
|                                       | Application rate and length of confinement |               |                 | 54 p.p.m. (10-percent concentration) 1 week | 108 p.p.m. (10-percent concentration) 1 week | No application - controls 1 week | 36 p.p.m. (5-percent concentration) 1 week | 54 p.p.m. (5-percent concentration) 1 week | 45 p.p.m. (10-percent concentration) 1 week | 45 p.p.m. (5-percent concentration) 1 week | 45 p.p.m. (3.33-percent concentration) 1 week | 45 p.p.m. (2.5-percent concentration) 1 week | No application - controls |

1/ Sampling periods correspond with those in tables 8 and 9.

Table 12. -- Comparative abundance of species of stored-grain insects found in samples of wheat taken from test bins, 1955-56

|  | d                | Proportion of | species present in samples taken during | ent in sample      | Ø                     | 6       |                        |
|--|------------------|---------------|---|--------------------|-----------------------|---------|------------------------|
| Source of samples and species of insects $\frac{1}{2}$ | 1955-56          |               | 1956                                    | 95                 |                       | insects | Percentage<br>of total |
|  | December-January | February-     | May-July                                | August-<br>October | November-<br>December | present |                        |
|  | Percent          | Percent       | Percent                                 | Percent            | Percent               | Number  | Percent                |
| Tests in treated series                                | -                | c             | c                                       | \<br>\<br>\        | c                     | -       |                        |
| Lesser grain borer.                                    | 08.2             | 86.)          | 0                                       | 76.2               | 0                     | ⇒ €     | 86.2                   |
| Saw-toothed grain beetle                               | 1,9              | 13.6          | 50.0                                    | 12.5               | 0                     | 2       | 7.5                    |
| Dermestids 3/  | 0                | 0             | 50.0                                    | 6.3                | 0                     | 2       | 2.1                    |
| Total number 4   | (45)             | (22)          | (2)                                     | (16)               | (0)                   | 94      |                        |
| Tests in control series                                |                  |               |   |                    |                       |         |                        |
| Lesser grain borer,                                    | 0                | 2.5           | 0                                       | 4.3                | 0                     | 22      | 200                    |
| Flat grain beetle2/                                    | 73.3             | 75.7          | 70.2                                    | 6.79               | 76.8                  | 321     | 72.6                   |
| Saw-toothed grain beetle                               | 26.7             | 14.8          | 25.5                                    | 26.4               | 21.1                  | 76      | 21.3                   |
| Dermestids 3/  | 0                | 0             | 4.3                                     | Z.                 | 1.8                   | 77      | 6.                     |
| Indian-meal moth                                       | 0                | 0             | 0                                       | 2                  | 0                     | _       | 2,                     |
| Total number 4/  | (30)             | (169)         | (47)                                    | (140)              | (95)                  | 7/17    |                        |
|  |                  |               |   |                    |                       |         |                        |

August 1955.

2/ Probably a complex of 3 species: Laemophloeus pusillus (Scholin,), L. ferrugineus (Steph.), and L. turcicus Grouv.

3/ Trogoderma spp.

4/ Numbers in parentheses represent the total of insects found during each period, and are not percentages.

Table 13. -- Comparative abundance of the species of stored-grain insects found in samples of shelled corn taken from test bins, 1953-56

| 195     195  -55     1955  -55     1955     1955     1956     19 |   |
|--|---|
| Percent   Perc | 1953-54   |
| Percent   Perc | November- Fe  |
| 57.1         71.5         90.0         79.0         31.8         30.8         34.5         256           3.5         11.5         90.0         5.3         11.5         2.6         9.6           3.9         11.5         6.7         6.7         9.3         30.8         34.5         6.9           3.9         11.5         6.7         6.7         6.7         9.8         10.9         10.9           3.2         11.5         6.7         6.7         9.8         10.9 <td>Percent Percent</td>  | Percent Percent   |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$   | 7.2 100.0<br>0 0<br>26.2 0<br>0 0<br>1,8  |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$   | 1,.8<br>0 0<br>0 0<br>0 0<br>0 0<br>0 0   |
| 5.4         1.6         3.2         7.8         .6         6.0         .6         6.0         .6         .1         0         .1         0         .1         0         .1         0         .1         0         .1         0         .1         0         .1         0         .1         0         0         .1         1         0         0         .1         1         0<   | 0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0<br>0 |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$   | .7 14.3<br>0 0<br>66.2 0<br>14.9 12.8   |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$   | .2 1½,3<br>1,8 0<br>11,2 0<br>0 0   |
|  | 1.6 14.3<br>1.9 0<br>0 0<br>0 0<br>0 0<br>0 0<br>0 0<br>0 0<br>0 0<br>0 0<br>0 0            |

1/ The scientific names of the insects listed herein can all be found in U. S. Dept. Agr. Farmers' bulletin 1260 (rev.), August 1955.
2/ Probably a complex of 3 species: Laemophloeus pusillus (Scholh.), L. ferrugineus (Steph.), and L. turcicus Grouv.
3/ Trogoderma spp.
1/ Numbers in parentheses represent the total of insects found during each period, and are not percentages.

#### FINDINGS

Although the studies with methoxychlor as a protective treatment have not been concluded, the following points were evident in the tests reported herein:

- 1. Methoxychlor dusts protected stored wheat and shelled corn from insect infestation over long periods of storage.
- 2. Application rates of 54 p.p.m. and above were highly effective on shelled corn for 2 full summer seasons after the year in which they were applied; rates of 36 p.p.m. and above were highly effective on shelled corn for 1 full summer season; and rates of 5 p.p.m. and above were apparently effective on wheat for 1 full summer season.
- 3. Uneven distribution of the protective dust in shelled corn resulted when distribution was dependent upon the action of the elevator auger and the cascading of the corn into the bin.
- 4. The actual residues on wheat following the application of dusts ranged from 45 to 80 percent, and those on shelled corn were about half the intended rate.
- 5. The depletion of the residues in the test grain was very gradual over the periods of observation.
- 6. The residues on shelled corn apparently lost none of their insecticidal effectiveness with age.
- 7. The residues in milling products occurred largely in the feed fractions with relatively small amounts in the flours.
- 8. Residues in flour were not destroyed by being baked in bread, although some reduction occurred.
- 9. A moisture content of 11.5 percent or less in wheat, or of 13.4 percent or lower in shelled corn, did not affect the performance of the protective dusts.
- 10. The commercial grade of wheat or shelled corn was not affected by application of protective dusts.





